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HIGH-TEMPERATURE TENSILE AND STRESS-RUPTURE PROPERTIES OF SOME ALLOYS IN THE TUNGSTEN-MOLYBDENUM SYSTEM

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SUMMARY

Three alloys consisting of 10, 25, and 50 weight percent molybdenum in tungsten were evaluated at high temperatures, 2500° to 4400° F, to determine their tensile properties. Results showed the 10 and 25 weight percent alloys have higher tensile strength than unalloyed tungsten at 2500° and 3000° F and equivalent strength to about 3500° F. The 50 weight percent alloy has strength equivalent to unalloyed tungsten from 2500° to 3200° F. Unalloyed tungsten has higher strength than the alloys above 3500° F. The results of stress-rupture tests for the 50 weight percent alloy are presented for the temperature range of 2500° to 3500° F in the as-worked and fully annealed conditions.

INTRODUCTION

The need for materials to withstand the very high temperatures encountered in solid-propellant rocket motors has stimulated interest in tungsten-molybdenum alloys. Although unalloyed tungsten is a very promising material for this application, its high density and poor fabricability are major disadvantages. Molybdenum additions to tungsten decrease density and are reported to improve machinability (ref. 1). Unfortunately, these benefits are achieved at the expense of lowering the melting point below that of unalloyed tungsten, with consequent reduction of strength at very high temperatures.

Mechanical properties of powder-metallurgy tungsten-molybdenum alloys at temperatures up to about 2800° F have previously been reported by Bückle (ref. 2). Similarly, Semchyshen (ref. 3) has reported tensile properties of several arc-melted tungsten-molybdenum alloys at temperatures up to 2400° F. The only available data for temperatures above 3000° F appear to be those of Foyle (ref. 4), who investigated tensile properties of arc-melted tungsten-base alloys containing 10 and 25 weight percent molybdenum, and Lake, Brezynyak, and Doble (ref. 5), who investigated tensile properties of arc-cast tungsten-base alloys containing 5 and 15 weight percent molybdenum.

This investigation was undertaken to determine the high-temperature tensile properties of powder-metallurgy tungsten-molybdenum alloys at temperatures up to 4400° F. For this purpose, sintered and swaged bars of alloys containing 10, 25, and 50 weight percent molybdenum were procured from a commercial vendor and were tensile tested at temperatures from 2500° to 4400° F. The 50 weight percent molybdenum in tungsten alloy was stress-rupture tested at 2500°, 3000°, and 3500° F.

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MATERIALS, APPARATUS, AND PROCEDURE

Materials

The materials evaluated were unalloyed tungsten (ref. 6) and tungsten plus 10, 25, and 50 weight percent molybdenum with the following chemical analysis:

Alloy composition	Constituent elements						
	Weight percent	ppm					
	Mo	C	N	O	Fe	Cr	Si
90% W - 10% Mo	10.09	7	28	4.3	600	10	20
75% W - 25% Mo	24.87	9	15	4.5	635	10	20
50% W - 50% Mo	49.86	13	25	156	850	20	20

The materials were received as 1/2-inch-diameter swaged bars and were evaluated in the as-received condition. In addition, some of the tungsten plus 50 weight percent molybdenum specimens were fully recrystallized at 3550° F for 1 hour prior to rupture testing. Photomicrographs of the as-received bars are shown in figures 1(a), (b), and (c), and the fully recrystallized bar is shown in figure 1(d).

Apparatus

The short-time tensile test apparatus and procedure used in this investigation are described in reference 6. The stress-rupture equipment consisting of a loading frame, vacuum chamber, vacuum pumping system, and power supply is shown in figure 2. The unit consists of a conventional frame to which is attached the vacuum chamber, a lever system for application of the dead weight load, and a control panel. A 5:1 lever arm was used to apply the load on the specimen.

The test chamber is a water-cooled stainless-steel vacuum chamber 10 inches in diameter by 20 inches in inside height, which is split longitudinally and hinged at one side, as shown in figure 2. Flanged openings are provided at the rear of the chambers for pumping and at the top for the admission of the specimen and thermocouple. Vacuum-tight seals, which are electrically isolated, are provided at the rear of the chamber for the water-cooled electrodes. Pull rods enter the top and bottom of the chamber through sleeves welded to the end plates. These rods provide a sliding seal by means of neoprene O-rings, which are seated in grooves in the rods and are compressed only enough to ensure a reliable vacuum seal.

The chamber is evacuated by a single-stage mechanical forepump rated at 30 cubic feet per minute (13 liters/sec) backing a water-cooled three-stage fractionating oil diffusion pump having a maximum pumping speed of 300 liters per second. Baffles, cooled by liquid nitrogen, are placed between the diffusion pump and the chamber to minimize back-diffusion of oil vapors to the chamber. Pressure is measured with a cold cathode ionization gage. Although the test chamber can be evacuated to 10^{-6} millimeter of mercury, outgassing of the specimen and furnace, on heating, causes the pressure to rise. Consequently, in most tests, the pressure at the beginning of the test was in the 10^{-4}-millimeter-mercury range and gradually decreased to the 10^{-5}-millimeter-mercury range.

Details of the heater assembly are shown in figure 3 and are described in reference 7. The power supply consists of a voltage regulator, capable of controlling the voltage to within 0.1 percent of the desired voltage, a motorized variable transformer for power control, and a water-cooled welding transformer with a 14-volt and 3000-ampere output. Air-cooled copper bus bars connect the transformer to the water-cooled electrodes which enter through the rear of the chamber.

<u>Specimens, grips, and loading fixtures</u>. - The design of the specimen is shown in figure 4. The grips and loading fixtures used in this investigation are described in reference 7.

<u>Temperature measurement and control</u>. - Specimen temperature was measured with a tungsten-molybdenum thermocouple spotwelded to the surface of the specimen at the midpoint of the gage length as described in reference 7. However, since these thermocouples become unstable at high temperatures with time, temperature control was not possible with thermocouples for the long-time stress-rupture tests. Thus it was necessary, upon attaining the test temperature, to depend solely on the voltage regulator to maintain the test temperature. By using a calibrated optical pyrometer sighted on a black-body hole, it was found that the specimen temperature did not vary more than ±10° F at 3500° F after 50 hours, indicating that voltage regulation was a satisfactory means of attaining temperature control.

Procedure

After the thermocoupled specimen had been installed in the load train, the chamber was evacuated to pressure in the range 10^{-4} to 10^{-5} millimeter of mercury. The specimen was then slowly heated to the test temperature (in about 45 min.) and allowed to soak for about 15 minutes to ensure equilibrium in the system. In the tensile tests, specimens were loaded to slightly beyond the 0.2 percent offset yield point at a constant crosshead speed of 0.005 inch per minute, after which the crosshead speed was increased to 0.05 inch per minute until fracture. For the stress-rupture tests, loading commenced by applying dead weights to the pan on the lever arm until the desired stress on the specimen had been attained. Rupture life was indicated by a running time meter that stopped when rupture occurred.

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RESULTS AND DISCUSSION

Comparison of High-Temperature Tensile Strength of As-Received Tungsten-Molybdenum Alloys

The results of the high-temperature tensile tests of the as-received materials are presented in table I and figure 5. The data for unalloyed tungsten, as noted in table I, were reported previously and showed a considerable difference in tensile strength below 3500° F for the several lots of commercial tungsten rods that were evaluated. This difference was, in all probability, due to differences in the amount of retained cold-work resulting from the fabrication history of these materials. Additional data from other sources and unpublished NASA data indicate that the scatter band for unalloyed tungsten should be weighted to the low side at 2500° and 3000° F, and it is so presented in figure 5.

In the temperature range from 2500° to about 3200° F, all three alloys of tungsten containing molybdenum have tensile strengths that are equivalent or superior to those of unalloyed tungsten. For example, the tungsten plus 25 percent molybdenum alloy (the strongest alloy) has a tensile strength of 30,000 pounds per square inch at 3000° F, whereas unalloyed tungsten ranges from 17,000 to 24,000 pounds per square inch. At this temperature, the tungsten plus 10 percent molybdenum alloy has a tensile strength of 28,000 pounds per square inch, and the tungsten plus 50 percent molybdenum alloy has a tensile strength of 20,000 pounds per square inch. At temperatures above 3200° to 3300° F these alloys lose their strength advantage over unalloyed tungsten, and are weaker above 3500° F.

Since weight is a very important consideration in selection of materials for aerospace applications, it is of interest to compare the tungsten-molybdenum alloys on a strength-to-weight basis. At temperatures at which their ultimate tensile strength is just equal to that of unalloyed tungsten, weight savings of about 25, 12, and 5 percent, respectively, would be achieved by substitution of the 50, 25, and 10 weight percent molybdenum alloys for unalloyed tungsten. Figure 6 compares the tensile strength-to-density ratio against temperature for unalloyed tungsten and the tungsten-molybdenum alloys. Comparison of figure 6 with figure 5 emphasizes the advantages of the alloys at temperatures up to about 3300° F and the superiority of unalloyed tungsten above 3500° F. It is apparent from both figures 5 and 6 that molybdenum is not a very potent strengthener in tungsten and that at very high temperatures the benefits of solid-solution strengthening are outweighed by the decrease in melting point as a result of alloying.

Ultimate tensile strength is plotted as a function of alloying composition for the various temperatures in figure 7(a), and the strength-to-density ratio is plotted against composition in figure 7(b). Data for unalloyed molybdenum are taken from reference 8. The optimum composition at 2500° and 3000° F is about 75 percent tungsten and 25 percent molybdenum where the strength is at a maximum. On an atomic percent basis, this composition is very close to the 50 percent tungsten - 50 percent molybdenum composition where maximum lattice strain is to be expected. The arc-melted tungsten-molybdenum alloys reported in references 4 and 5 are slightly stronger at 2500° and 3000° F than the powder-metallurgy alloys reported herein. At 3500° F and above, the ultimate tensile strengths are equivalent.

High-Temperature Ductility of Tungsten-Molybdenum Alloys

Ductility, as measured by the reduction of area of the fractured specimens, is plotted as a function of temperature for unalloyed tungsten and the three tungsten alloys in figure 8. All three tungsten-molybdenum alloys exhibited a minimum in ductility at high temperatures and in this respect behaved similarly to unalloyed powder-metallurgy tungsten. This minimum occurs at approximately 3500° F, whereas the ductility minimum for unalloyed tungsten occurs at about 4000° F.

Comparison of Stress-Rupture Properties of As-Received and Fully Annealed 50 Percent Tungsten - 50 Percent Molybdenum Alloy

Stress-rupture properties of the 50 percent tungsten - 50 percent molybdenum alloy were determined at 2500°, 3000°, and 3500° F in the as-received and fully annealed conditions. Sufficient material was not available for similar evaluation of the other alloy compositions. The results are tabulated in table II and are plotted in figure 9. Some of the specimens were fully recrystallized at 3500° F for 1 hour, prior to testing, in order to minimize any structural differences within the bar stock. The results of strengthening due to cold-work may be seen in the 2500° F curves of figure 9. For example, the rupture life of the as-received material under a stress of 10,000 pounds per square inch is almost 3400 minutes, but after annealing the rupture life decreases to less than 300 minutes. At 3000° F the as-received material has greater rupture strength at stresses in excess of 4500 pounds per square inch where the time at temperature is short enough to preclude the annealing out of the cold-work. The alloy becomes fully annealed at 3500° F even after short periods of time, as evidenced by the coincidence of the as-received curve with the fully annealed curve.

Recrystallization of Tungsten-Molybdenum Alloys

In order to more fully characterize the tungsten-molybdenum system, recrystallization temperatures were determined and are tabulated in table III. The recrystallization temperature, as used here, is defined as the minimum temperature for which in 1 hour:

(1) The structure is at least 50-percent recrystallized when examined metallographically in both longitudinal and transverse directions at a magnification of 100.

(2) The drop in hardness is at least two-thirds of the total drop from the as-worked to the fully annealed condition.

For all three alloys, the recrystallization temperature was about 3000° F for the 1/2-inch-diameter swaged bars that were evaluated.

SUMMARY OF RESULTS

The following results were obtained from an investigation of the high-temperature tensile properties of three alloys consisting of 10, 25, and 50 weight percent molybdenum in tungsten and the stress-rupture properties of a 50 weight percent molybdenum in tungsten alloy:

1. The 10 and 25 weight percent molybdenum-tungsten alloys have greater strength than unalloyed tungsten at 2500° and 3000° F and comparable strength to 3500° F.

2. The 50 weight percent alloy has comparable strength to unalloyed tungsten to about 3200° F. Because of its considerably lower density, significant weight reductions, up to about 25 percent, would result from substitution of this alloy for unalloyed tungsten where feasible.

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3. One- and ten-hour rupture strengths of the recrystallized 50 percent tungsten - 50 percent molybdenum alloy are as follows:

Temperature, °F	Time, hr	
	1	10
2500	[a]13,000 psi	8800 psi
3000	6,000 psi	[a]4000 psi
3500	3,400 psi	2000 psi

[a]Extrapolated values.

Lewis Research Center
National Aeronautics and Space Administration
Cleveland, Ohio, January 24, 1962

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TABLE I. - TENSILE PROPERTIES OF UNALLOYED TUNGSTEN AND THREE TUNGSTEN-MOLYBDENUM ALLOYS

Temperature, °F	Ultimate tensile strength, psi	Elongation, percent	Reduction of area, percent
Pure W[a]			
2500	37,800 52,200	25 25	87 94
3000	20,250 29,900	51 22	81 79
3500	9,180 11,500	35 23	42 28
4000	5,820 6,890	15 21	16 24
4400	4,080 4,710	23 22	30 26
90% W - 10% Mo			
2300	49,000	26	86
2500	44,600	21	98
3000	28,300	25	84
3500	10,300	27	39
4000	4,550	26	42
4400	2,660	35	56
75% W - 25% Mo			
2500	46,350	20	63
3000	29,400	15	42
3500	8,200	11	18
4000	4,500	15	25
50% W - 50% Mo			
2500	36,200	22	68
3000	20,400	15	42
3500	6,500	11	16
4000	4,300	10	27
4400	1,900	14	29

[a] Data taken from ref. 6.

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TABLE II. - STRESS-RUPTURE PROPERTIES OF 50 WEIGHT PERCENT

MOLYBDENUM-TUNGSTEN ALLOYS

Temperature, °F	Stress, psi	As-received			Recrystallization at 3550° F for 1 hr		
		Rupture life, min	Elongation, percent	Reduction of area, percent	Rupture life, min	Elongation, percent	Reduction of area, percent
2500	18,000	211	17	33	----	--	--
	12,500	1180	--	--	----	--	--
	10,000	3356	19	31	290	6	23
	6,000	----	--	--	5369	--	--
3000	10,000	42	--	--	2	8	18
	7,000	103	14	22	---	--	--
	6,000	159	11	18	76	11	19
	5,000	---	--	--	173	--	--
3500	5,000	11	15	21	8	8	14
	2,500	191	10	19	---	--	--
	3,000	----	--	--	100	--	--
	1,500	1911	12	23	---	--	--

TABLE III. - RECRYSTALLIZATION DATA FOR THREE

TUNGSTEN-MOLYBDENUM ALLOYS

Alloy composition	Recrystallization temperature, °F	As-received hardness Vickers, 10-kg load	Fully annealed hardness Vickers, 10-kg load	Percent of drop in hardness at recrystallization temperature
90% W - 10% Mo	3000	422	335	67.8
75% W - 25% Mo	3000	374	304	61.4
50% W - 50% Mo	3000	301	223	69.2

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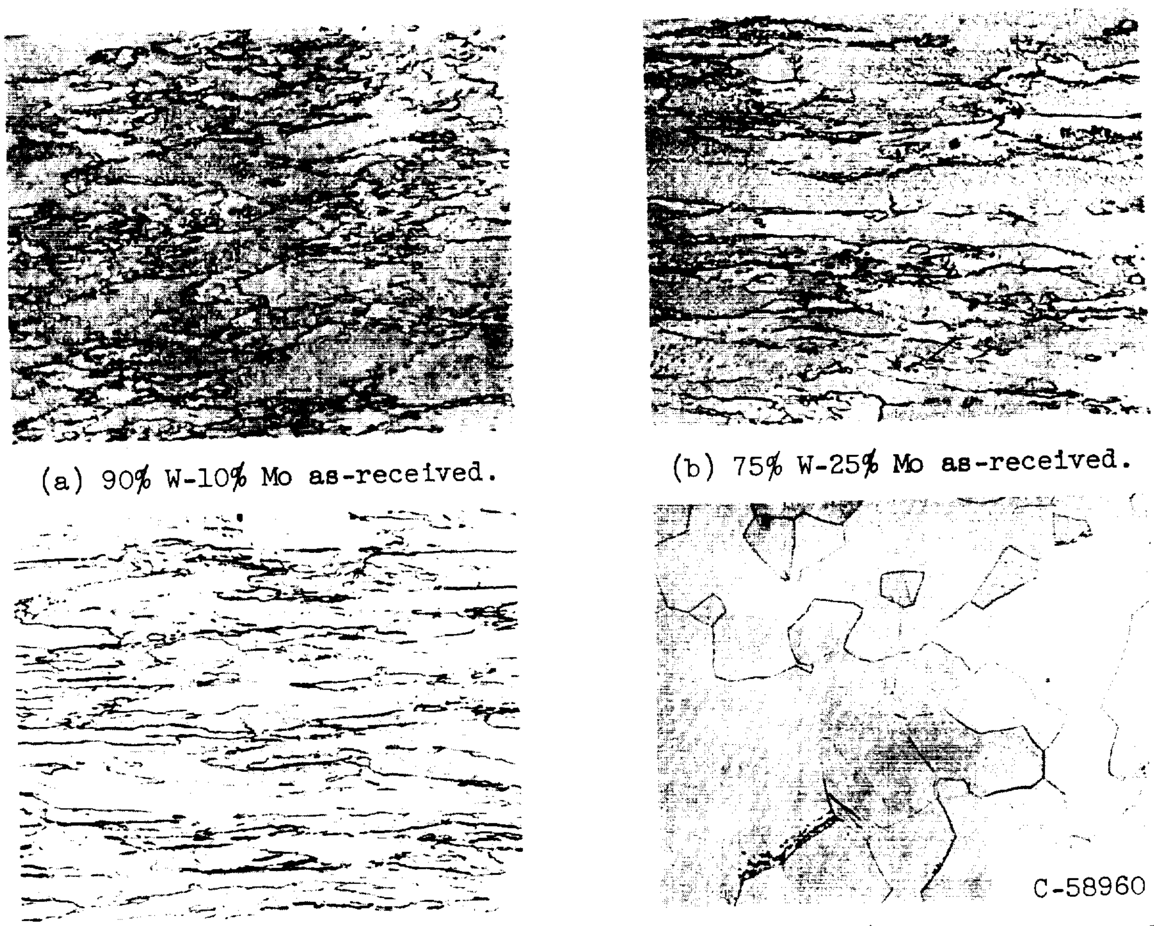

(a) 90% W-10% Mo as-received.

(b) 75% W-25% Mo as-received.

(c) 50% W-50% Mo as-received.

(d) 50% W-50% Mo recrystallized at 3550° F for 1 hour.

Figure 1. - Microstructure of as-received tungsten-molybdenum alloys and of 50 percent tungsten-50 percent molybdenum alloy fully recrystallized. Longitudinal section; X 250.

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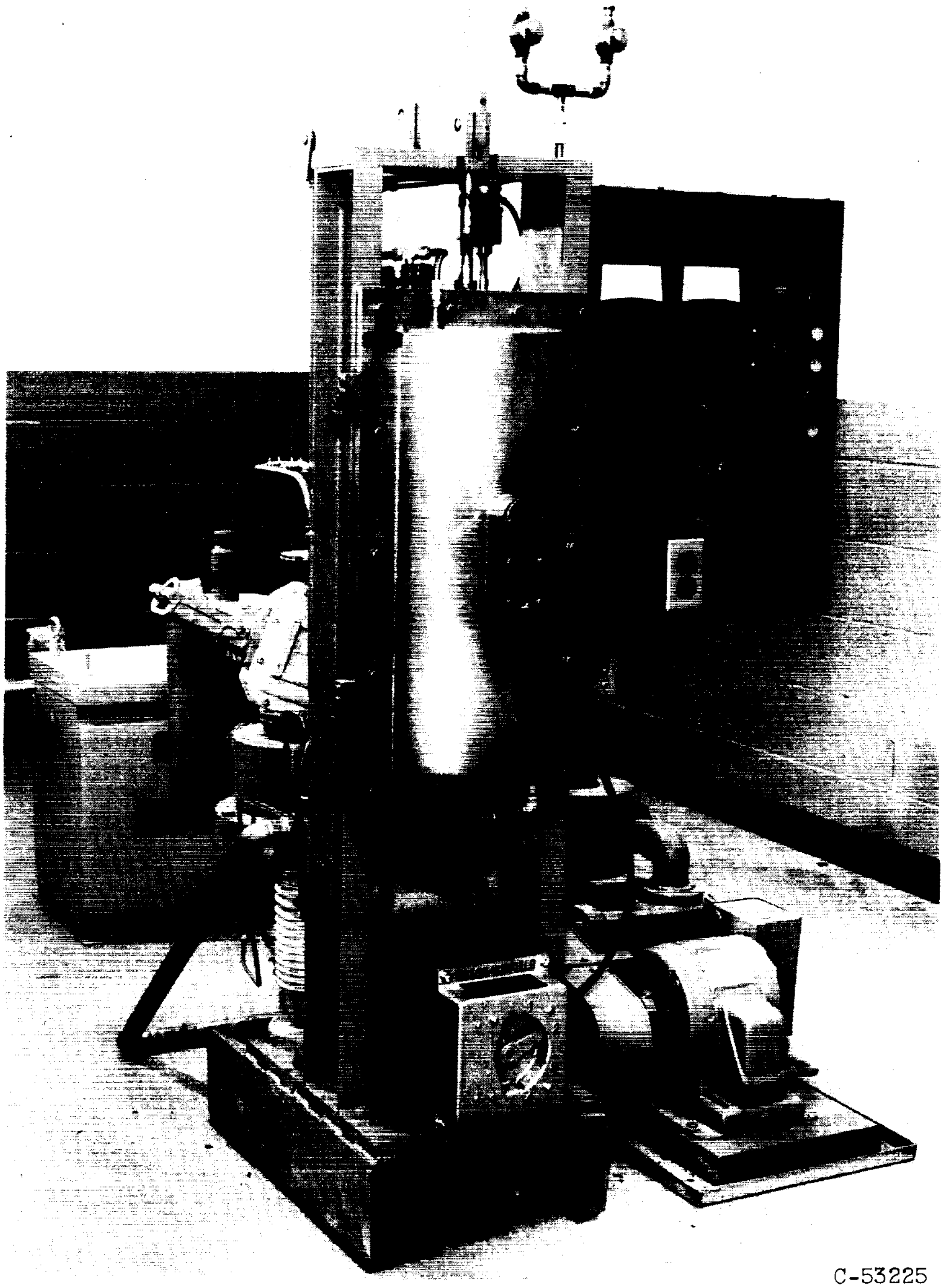

Figure 2. - High-temperature stress-rupture equipment.

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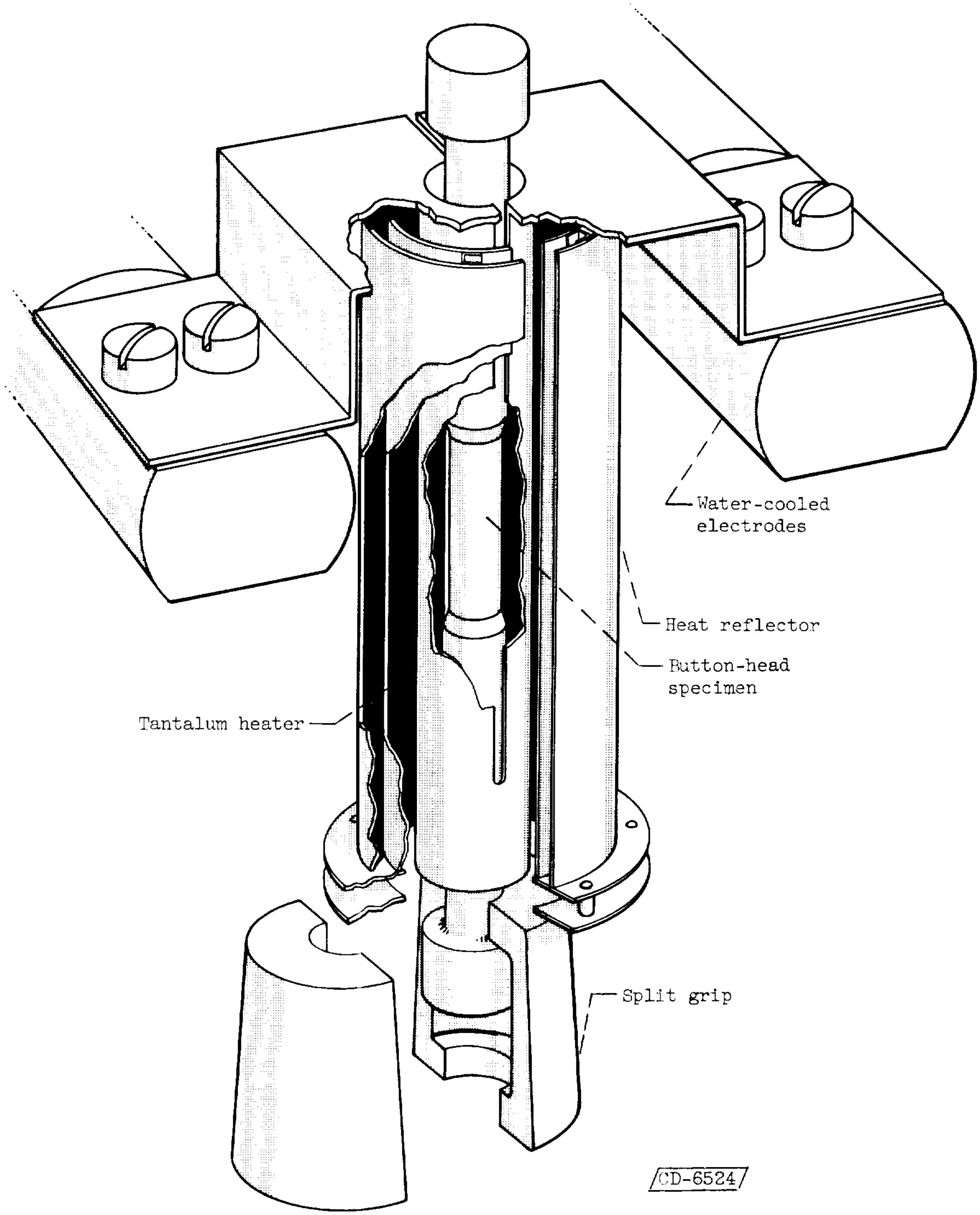

Figure 3. - Heater assembly for high-temperature tests.

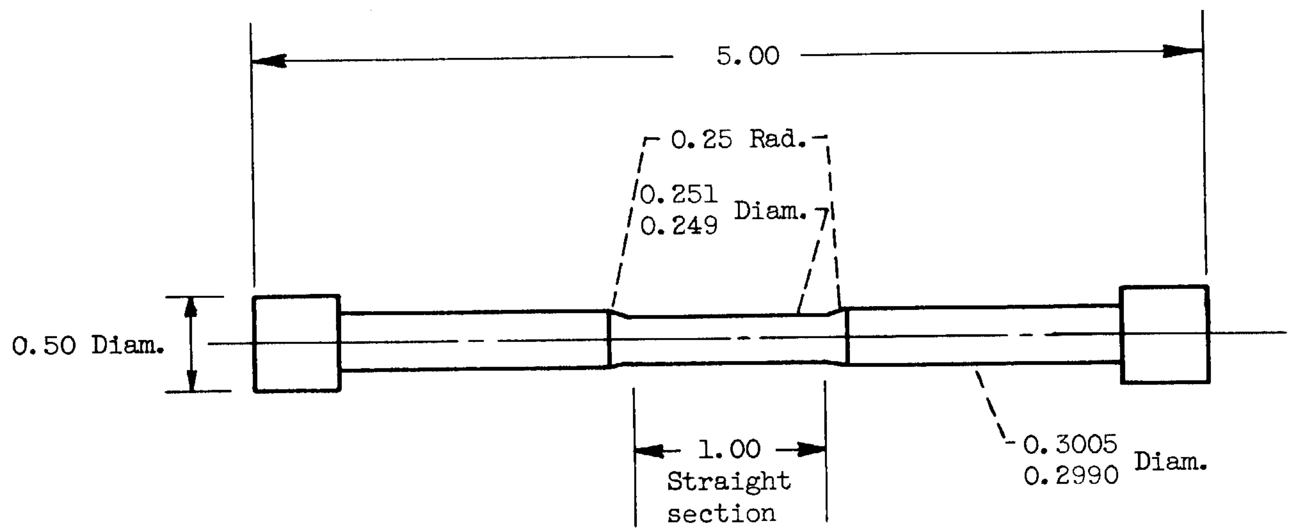

Figure 4. - Button-head tensile specimen. (All dimensions in inches.)

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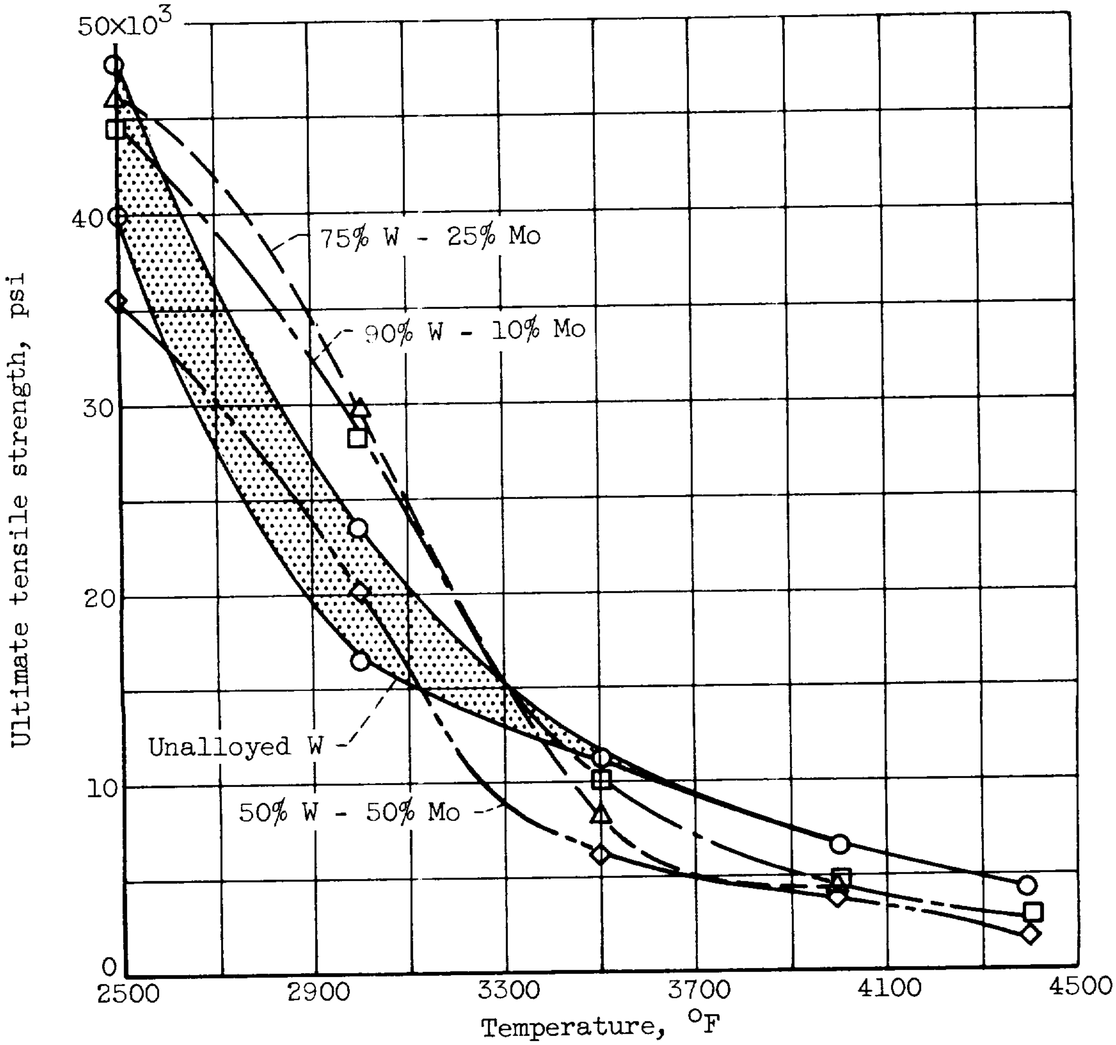

Figure 5. - Ultimate tensile strength of unalloyed tungsten and three tungsten-molybdenum alloys.

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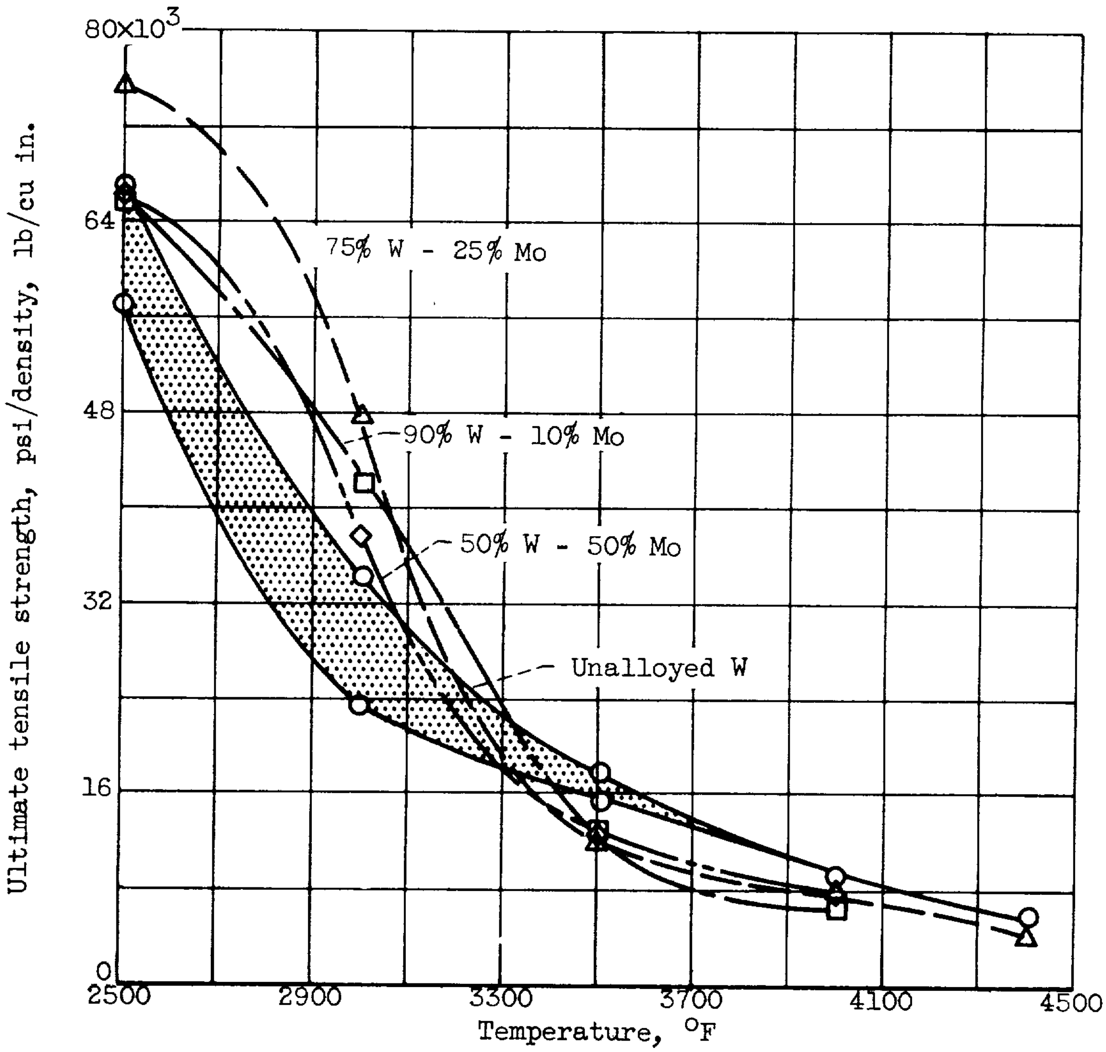

Figure 6. - Strength-to-weight ratio of unalloyed tungsten and three tungsten-molybdenum alloys.

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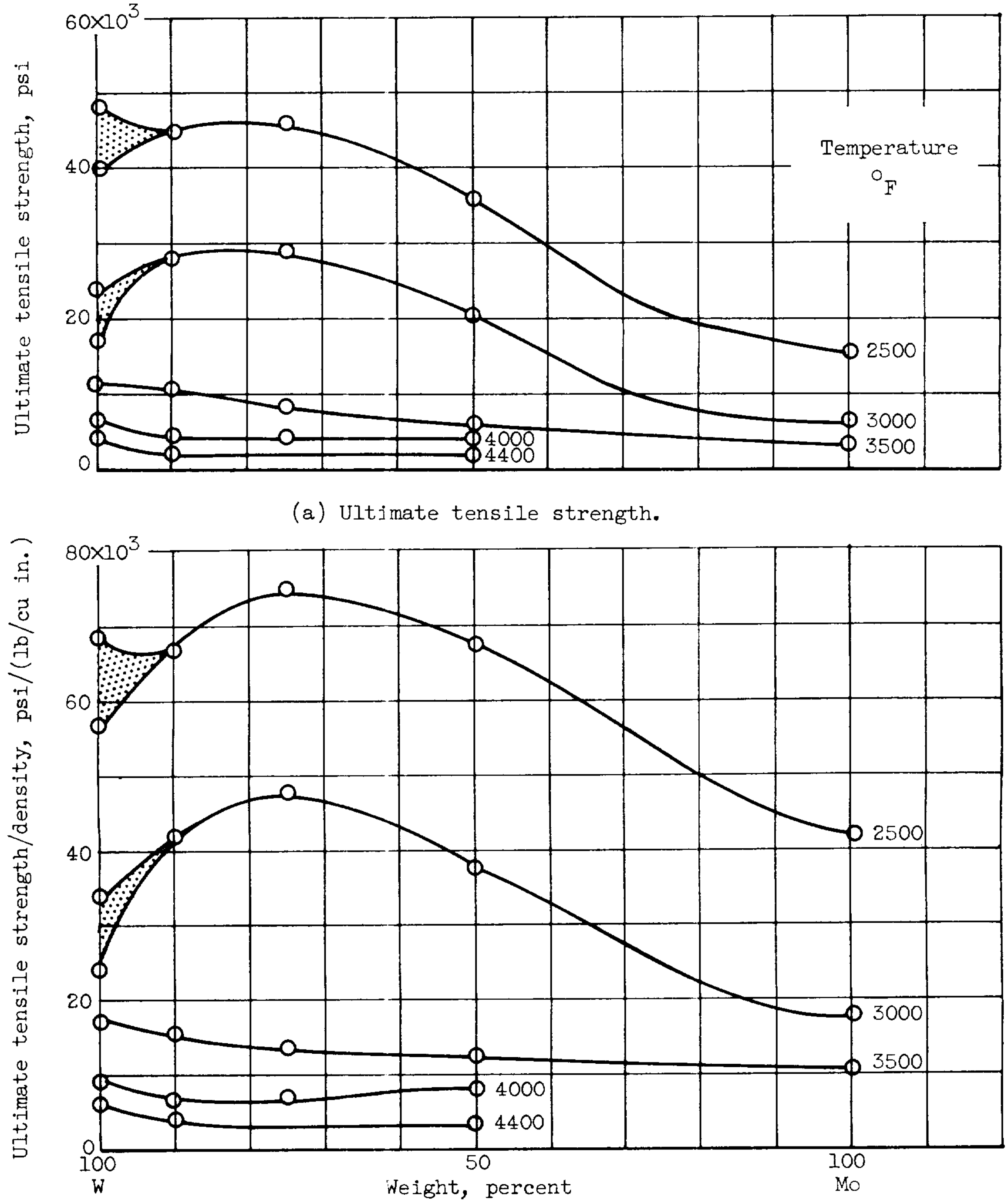

(b) Strength-to-weight ratio.

Figure 7. - Effect of alloying tungsten with molybdenum on ultimate tensile strength and the strength-to-weight ratio.

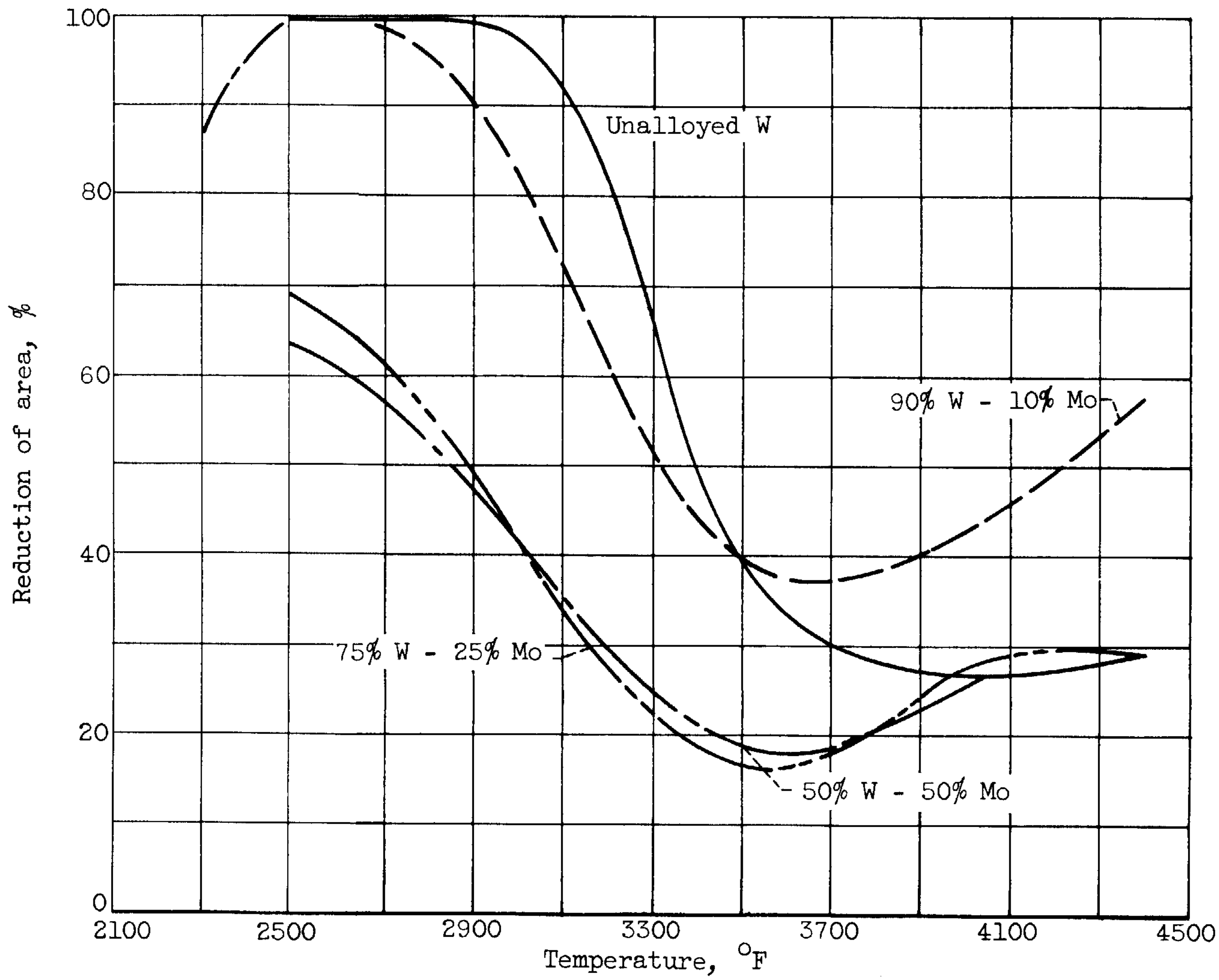

Figure 8. - Reduction of area resulting from tensile tests of unalloyed tungsten and three tungsten-molybdenum alloys.

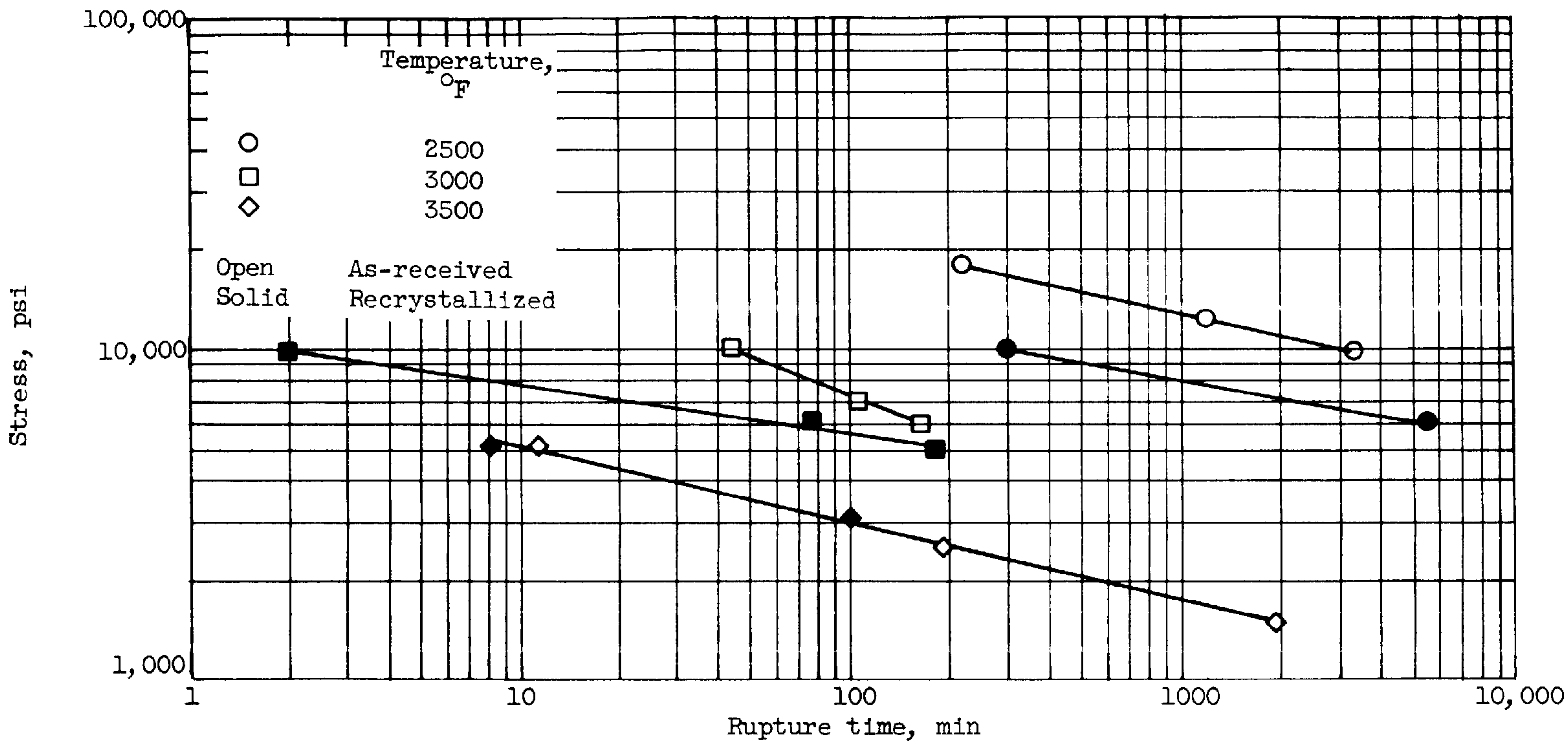

Figure 9. - Stress-rupture properties of 50 percent tungsten - 50 percent molybdenum alloy.

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